PATENT ABSTRACTS OF JAPAN

(11)Publication number : 07-291804

(43)Date of publication of application : 07.11.1995

(51)Int.Cl. A01N 25/12
A01N 25/08
A01N 25/22
A01N 43/40

(21)Application number : 06-088846

(22)Date of filing : 26.04.1994

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(54) WATER-SOLUBLE GRANULAR AGROCHEMICAL

(57)Abstract:

PURPOSE: To obtain a water-soluble granular agrochemical improved in storage stability of its active component without reducing the ready water solubility.

CONSTITUTION: This water-soluble granular agrochemical contains (A) a water- soluble active component, e.g. an active component having ≥0.01g/ml, preferably ≥0.1g/ml water solubility at 20°C and (B) a water-soluble weakly basic substance, preferably a substance having ≥0.05 g/ml water solubility at 20°C and exhibiting pH 7.0 to 9.0 at 25°C in the form of an aqueous solution, e.g. sodium thiosulfate, disodium hydrogen phosphate, sodium acetate, sodium carbonate or sodium bicarbonate in a ratio of 5 to 95wt.% (A) and 5 to 94wt.% (B). As the component (A), cartap hydrochloride, nitenpyram, allethrin, acephate, ESP (oxydeprofos), vamidothion, DEP (trichlorfon), validamycin A, diquat and bialaphos are exemplified.

LEGAL STATUS

[Date of request for examination] 31.01.2001

[Date of sending the examiner's decision of rejection]

[Kind of final disposal of application other than the examiner's decision of rejection or application converted registration]

[Date of final disposal for application]

[Patent number]

[Date of registration]

[Number of appeal against examiner's decision of rejection]

[Date of requesting appeal against examiner's decision of rejection]

[Date of extinction of right]

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CLAIMS

[Claim(s)]
[Claim 1] A water-soluble granule characterized by containing a water-soluble agricultural-chemicals active ingredient and water-soluble weak base nature material.
[Claim 2] A water-soluble granule according to claim 1 whose solubility to water [in / in a water-soluble agricultural-chemicals active ingredient / 20 degrees C] is a 0.01g [/ml] or more water solubility agricultural-chemicals active ingredient.
[Claim 3] A water-soluble granule according to claim 1 whose water-soluble agricultural-chemicals active ingredient is NITEMUPIRAMU.
[Claim 4] A water-soluble granule according to claim 1 to 3 whose solubility to water [in / in water-soluble weak base nature material / 20 degrees C] is 0.05g/ml or more and whose pH of an aqueous solution in 25 degrees C is the water-soluble weak base nature material of about 7.0-9.0.
[Claim 5] A water-soluble granule according to claim 4 whose solubility to water in 20 degrees C is 0.05g/ml or more and whose pH of an aqueous solution in 25 degrees C is one sort or two sorts or more of water-soluble weak base nature material with which water-soluble weak base nature material of about 7.0-9.0 is chosen from a group which consists of a sodium thiosulfate, disodium hydrogen-phosphate, sodium acetate, a sodium carbonate, and sodium bicarbonate.
[Claim 6] A water-soluble granule according to claim 1 characterized by containing water-soluble weak base nature material for a water-soluble agricultural-chemicals active ingredient about 15 to 94% of the weight about five to 95% of the weight to the water-soluble granule whole quantity.

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PRIOR ART

[Description of the Prior Art] Nitenpyram (nitenpyram, (E)-N-(6-chloro-3-pyridyl methyl)-N - ethyl-N'-methyl-2-nitro vinylidene diamine) is an insecticide for agriculture which has the outstanding insect-pest-control capacity. It can mix with other insecticides or a germicide, it can mix with mineral powder, water-soluble support, an extending agent, etc., and can use as various solid preparations, such as powder material, a granule, water dispersible powder, water soluble powders, and a granule, (JP,2-171,A). It is the pharmaceutical preparation which whose water-soluble agricultural-chemicals granule is a granule which dissolves in water completely easily, and was excellent in it in respect of the simplicity of the ease of handling, the ease of measuring, dust prevention, and use. For example, the outstanding Tempe Lamb content water solubility granule with which the lactose is blended as an extending agent is proposed (JP,6-92803,A). Furthermore, decomposition of NITEMUPIRAMU in a water-soluble granule was controlled more, and development of the water-soluble granule stabilized more was desired.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Industrial Application] This invention relates to the water-soluble stable granule containing water-soluble agricultural-chemicals active ingredients, such as nitenpyram. The pharmaceutical preparation of this invention is used for the purpose of pest control in the field of agricultural.
[0002]
[Description of the Prior Art] Nitenpyram (nitenpyram, (E)-N-(6-chloro-3-pyridyl methyl)-N - ethyl-N'-methyl-2-nitro vinylidene diamine) is an insecticide for agriculture which has the outstanding insect-pest-control capacity. It can mix with other insecticides or a germicide, it can mix with mineral powder, water-soluble support, an extending agent, etc., and can use as various solid preparations, such as powder material, a granule, water dispersible powder, water soluble powders, and a granule, (JP,2-171,A). It is the pharmaceutical preparation which whose water-soluble agricultural-chemicals granule is a granule which dissolves in water completely easily, and was excellent in it in respect of the simplicity of the ease of handling, the ease of measuring, dust prevention, and use. For example, the outstanding Tempe Lamb content water solubility granule with which the lactose is blended as an extending agent is proposed (JP,6-92803,A). Furthermore, decomposition of NITEMUPIRAMU in a water-soluble granule was controlled more, and development of the water-soluble granule stabilized more was desired.
[0003]
[Problem(s) to be Solved by the Invention] This invention aims at offering the water-soluble new agricultural-chemicals granule which can control decomposition of water-soluble agricultural-chemicals active ingredients, such as NITEMUPIRAMU. It aims at offering the granule which raised the conservation stability of a water-soluble active ingredient, without spoiling especially soluble [to the water which is the feature of a water-soluble granule].
[0004]
[Means for Solving the Problem] this invention persons found out that a water-soluble agricultural-chemicals granule with also unexpectedly stable nitenpyram was obtained by blending and carrying out wet agglomeration of the water-soluble weak base nature material to nitenpyram, as a result of inquiring wholeheartedly, in order to solve the above-mentioned technical problem. Furthermore, this invention persons came to complete this invention, as a result of finding out that a water-soluble stable agricultural-chemicals granule is obtained and repeating examination further, even if applied not only to NITEMUPIRAMU but to other water-soluble agricultural-chemicals active ingredients. Namely, a water-soluble granule characterized by this invention containing (1) water solubility agricultural-chemicals active ingredient and water-soluble weak base nature material, (2) A water-soluble granule given in ** (1) term given solubility to water [in / in a water-soluble agricultural-chemicals active ingredient / 20 degrees C] is a 0.01g [/ml] or more water solubility agricultural-chemicals active ingredient, (3) A water-soluble granule given in ** (1) term a water-soluble given agricultural-chemicals active ingredient is NITEMUPIRAMU, (4) Solubility to water [in / in water-soluble weak base nature material / 20 degrees C] is 0.05g/ml or more, and pH of an aqueous solution in 25 degrees C A water-soluble granule given in about 7.0 ** (1) term - ** (3) term which is the water-soluble weak base nature material of 9.0, (5) Solubility to water in 20 degrees C is 0.05g/ml or more, and pH of an aqueous solution in 25 degrees C water-soluble weak base nature material of about 7.0-9.0 As opposed to a water-soluble granule given in ** (4) term which is one sort chosen from a group which consists of a sodium thiosulfate, disodium hydrogen-phosphate, sodium acetate, a sodium carbonate, and sodium bicarbonate, or two sorts or more, and (6) water-solubility granule whole quantity A water-soluble granule given in ** (1) term characterized by containing water-soluble weak base nature material for a water-soluble agricultural-chemicals active ingredient about 15 to 94% of the weight about five to 95% of the weight is offered.

[0005] A ** (1) term whose (6) water solubility agricultural-chemicals active ingredient is still more specifically a water-soluble stable agricultural-chemicals active ingredient under alkaline conditions, or a water-soluble granule given in ** (2) term, (7) A water-soluble agricultural-chemicals active ingredient Nitenpyram, allethrin, acephate, ESP (oxy-DEPUROPOSU), vamidothion, DEP (trichlorfon), A water-soluble granule given in ** (1) term which is one sort or two sorts or more of water-soluble agricultural-chemicals active ingredients chosen from a group which consists of validamycin A, diquat, and beer RAHOSU, (8) A water-soluble granule a ** (1) term which contains a surfactant further - given in ** (7) term, (9) A water-soluble granule given in ** (8) term a surfactant is [a given HLB value] about nine to 12 nonionic surfactant, (10) About nine to 12 nonionic surfactant Polyoxyethylene nonylphenyl ether, [an HLB value] Or a water-soluble granule given in ** (8) term characterized by containing a surfactant about 0.05 to 5% of the weight is offered to a water-soluble granule given in ** (9) term which is the block copolymerization object of ethyleneoxide and propylene oxide, and (11) water-solubility granule whole quantity.

[0006] As a water-soluble agricultural-chemicals active ingredient used for a water-soluble granule of this invention, about 0.01g /or more of solubility to water in 20 degrees C is preferably used for an about 0.1g [/ml] or more agricultural-chemicals active ingredient etc. ml, for example, and all agricultural-chemicals active ingredients, such as an insecticide, a germicide, and a herbicide, are used as this agricultural-chemicals active ingredient, for example. Also in these water-soluble agricultural-chemicals active ingredients, an unstable thing is not suitable for the bottom of alkaline conditions, and a water-soluble stable agricultural-chemicals active ingredient is suitable for the bottom of alkaline conditions. a water-soluble granule of this invention -- setting -- these water solubility agricultural-chemicals active ingredient -- one sort -- or two or more sorts can be used. Specifically as a water-soluble agricultural-chemicals active ingredient used for a water-soluble granule of this invention, the following etc. are mentioned.

Solubility(water) Melting point (degree C)

- Insecticide-nitenpyram (nitenpyram) >200 g/l 84 -85 allethrin (allethrin) 500 g/l Liquid acephate (acephate) 650 g/l 91-92ESP, oxy-DEPUROPOSU (oxydeprofos) Dissolution Liquid vamidothion (vamidothion) 4000 g/l 46-48DEP, trichlorfon (trichlorfon) 154 g/l (25 degrees C) 83-84-germicide-validamycin A (validamycin A) Easily dissolvable 135-herbicide-diquat (diquat) Also in a water-soluble agricultural-chemicals active ingredient of the 700 g/l beer RAHOSU (bialaphos) >1000 g/l above One sort or two sorts or more of water-soluble agricultural-chemicals active ingredients chosen from a group which consists of NITEMUPIRAMU, acephate, validamycin A, etc. are desirable, and NITEMUPIRAMU etc. is especially suitable.

[0007] Although especially water-soluble weak base nature material used for a water-soluble granule of this invention is not limited, a thing whose solubility to water in 20 degrees C is 0.05g/ml or more, for example and whose pH of an aqueous solution in 25 degrees C is about 7.0-9.0 is suitable for it. One sort or two sorts or more of things specifically chosen from a group which consists of a sodium thiosulfate, disodium hydrogen-phosphate, sodium acetate, a sodium carbonate, sodium bicarbonate, etc. are used. A method of an office, industrial use, a food additive, etc. can use anything, and grade of this water-soluble weak base nature material can be [a food additive etc.] scrupulous and use them also for existence of bound water further. A content of a water-soluble agricultural-chemicals active ingredient used for a water-soluble granule of this invention is usually about 5 - 80 % of the weight preferably about five to 95% of the weight to the granule whole quantity. A content of water-soluble weak base nature material used for a water-soluble granule of this invention is usually about 30 - 90 % of the weight preferably about 15 to 94% of the weight to the granule whole quantity. A water-soluble granule of this invention may contain water-soluble adjuvants, such as a surfactant, coloring matter, a binder, and an ultraviolet ray absorbent, further.

[0008] Especially as this surfactant, although not limited, a nonionic surfactant is used comparatively preferably. Especially a thing of about nine to 12 range has a desirable HLB value, in view of solubility and a point of a humid operation over water. specifically, the polyoxyethylene nonylphenyl ether (example: NP-85 (trade name), the Takemoto Fats-and-oils company make, etc.), block copolymerization objects of ethyleneoxide and propylene oxide, such as etc., example: -- new pole PE-64 (trade name) and Mitsuhiro -- Formation -- a shrine -- make, etc. are used. As this coloring matter, the cyanine green G (trade name) (Sumitomo Chemical [Co., Ltd.] Co., Ltd. make) etc. is specifically used. As this binder, a dextrin, polyvinyl alcohol, gum arabic, sodium alginate, a polyvinyl pyrrolidone, a glucose, cane sugar, a mannitol, a sorbitol, etc. are specifically used. A surfactant of a content in a water-soluble granule of other water-soluble adjuvants of these is usually 1.0 - 3.0% of the weight of a range preferably about 0.05 to 5% of the weight to the water-soluble granule whole quantity, coloring matter is usually about 0.01 - 0.1% of the weight of a range preferably about 0.01 to 0.5% of the weight, and a binder is usually preferably chosen about 0.5 to 10.0% of the weight in about 2.0 - 7.0% of the weight of the range.

[0009] A water-soluble granule of this invention can be manufactured according to a manufacturing method of the water-soluble usual granule. For example, it can manufacture using a spray drying method or the wet ********** ? Extrusion ?

method. In the case of a spray drying method, to pharmaceutical preparation solid content of the 100 weight sections, water of the about 50 to 1000 weight section is usually added, it dissolves, the powder granulation of the aqueous solution is carried out by spray drying, and a water-soluble granule is obtained. It dissolves in water of a water-soluble agricultural-chemicals active ingredient, water-soluble weak base nature material, and an amount of a degree which will fully dissolve other water-soluble adjuvants, such as a surfactant, if required, spray drying of this aqueous solution is carried out, and, specifically, a water-soluble granule is obtained. Or a water-soluble agricultural-chemicals active ingredient and water-soluble weak base nature material are dissolved in water of optimum dose, and after spray drying, other water-soluble adjuvants, such as a surfactant, may be added and you may mix. Moreover, you may use for a wet knockout granulation which mentions this spray drying article later. Obtained granulatio has a thing of the range of about 0.1-0.5mm preferably suitable for the particle diameter about 0.05-1.0mm. a case of a wet knockout granulation -- pharmaceutical preparation solid content of the 100 weight sections -- receiving -- usually -- the about one to 10 weight section -- it manufactures preferably using water of the about two to 5 weight section. If it is a water-soluble agricultural-chemicals active ingredient, water-soluble weak base nature material, and necessity, specifically, other water-soluble adjuvants, such as a surfactant, will be mixed to homogeneity with a mixer etc. Under the present circumstances, it is desirable to make it powder with moderate 100-micrometer or less degree by coarse grinding etc., when particle diameter of a formed element cannot be greatly mixed to homogeneity, and to make mixed actuation easy. Water of optimum dose is added to this mixture, and it scours with a mixer further even to a degree suitable for a knockout granulation of an after production process. This kneading object is dried after granulation with the usual knockout granulating machine. The particle size regulation of the granulation object is carried out as occasion demands, and a water-soluble desired granule is obtained. A particle size regulation is performed in the range which does not pass a screen of 300 micrometers of openings, but passes a 1,700-micrometer screen. Obtained granulatio has a thing of the range of about 0.5-5.0mm preferably suitable for the major axis about 0.5-10.0mm.

[0010] Moreover, bulk density of this granulatio usually has the desirable range of about 0.1-1.2g/ml, and its range of about 0.5-1.0g/ml is still more desirable. Thus, a water-soluble granule of this invention obtained does not have phytotoxicity substantially to vegetation, and it is harmless after the time of use, or use also to people or an animal, and can be used for safety. And the conservation stability of a water-soluble agricultural-chemicals active ingredient is raised by water-soluble granule of this invention, without spoiling soluble [to water which is the feature of a water-soluble granule]. a water-soluble granule of this invention -- usually -- water -- business -- the time -- dissolution dilution -- it is used by carrying out. Although it is not necessary to limit especially the dilution, its about (about 1g / 100ml - 1g / 2,000ml) about 100 to 2,000 times are usually desirable. And although irrelevance of this diluent changes with a water-soluble agricultural-chemicals active ingredient, an object noxious insect, object farmland, seasons, etc., it usually sprinkles preferably fields, such as a paddy field, a tea garden, and a kitchen garden, about 500-15000ml per ha of about 1000-10000ml of orchards etc., etc.

[0011]

[Example] Although an example, the example of reference, and the example of a trial are indicated below and this invention is explained more to it at details, this invention is not limited to these examples. In addition, especially % and the section that are used in here show weight % and the weight section altogether, as long as it is unstated.

[Example] Although an example, the example of a comparison, and the example of an experiment are indicated below and this invention is further explained to it at details, this invention is not limited to these examples. In addition, especially % and the section that are used in here show weight % and the weight section altogether, as long as it is unstated.

[Example 1] The nitenpyram 10 section and the sodium-thiosulfate 90 section were dissolved in the water of the 400 sections, the solid granulation of this aqueous solution was carried out with the spray dryer (the Okawara-ized ** machine, L-8), and the water-soluble granule containing nitenpyram 10% was obtained.

[Example 2] It considered as the disodium hydrogen-phosphate 90 section instead of the sodium-thiosulfate 90 section of an example 1, spray drying was carried out in the same procedure as an example 1, and the water-soluble granule containing nitenpyram 10% was obtained.

[Example 3] It considered as the sodium acetate 90 section instead of the sodium-thiosulfate 90 section of an example 1, spray drying was carried out in the same procedure as an example 1, and the water-soluble granule containing nitenpyram 10% was obtained.

[Example 4] It considered as the sodium-carbonate 90 section instead of the sodium-thiosulfate 90 section of an example 1, spray drying was carried out in the same procedure as an example 1, and the water-soluble granule containing nitenpyram 10% was obtained.

[0012]

[Example 5] It considered as the sodium bicarbonate 90 section instead of the sodium-thiosulfate 90 section of an example 1, spray drying was carried out in the same procedure as an example 1, and the water-soluble granule containing nitenpyram 10% was obtained.
[Example 6] The nitenpyram 10 section, surfactant new pole PE-64 The 1.0 sections, coloring matter cyanine green G The 0.15 sections, binder cane sugar The 5.0 sections, sodium bicarbonate After mixing the 83.85 sections enough, the water of the four sections was added and it kneaded with the kneading machine (the Kikusui factory, KM-1.5). This kneaded object was corned into cylinder-like granulatio with the knockout granulating machine (the Kikusui factory, RG-5M) using the screen of the diameter of 0.8mm. The water-soluble granule which dries the obtained granulatio at 60 degrees C for 1 hour, and contains nitenpyram 10% was obtained. EXTRUSION ?
[Example 7] It considered as the sodium acetate 10 section and the sodium bicarbonate 73.85 section at the change of the sodium bicarbonate 83.85 section of an example 6, and extruded and corned in the same procedure as an example 6, and the water-soluble granule containing nitenpyram 10% was obtained.
[The example 1 of reference] It considered as the lactose 90 section instead of the sodium-thiosulfate 90 section of an example 1, spray drying was carried out in the same procedure as an example 1, and the water-soluble granule containing nitenpyram 10% was obtained.
[The example 2 of reference] Sodium bicarbonate of an example 6 It considered as the lactose 83.85 section instead of the 83.85 sections, and extruded and corned in the same procedure as an example 6, and the water-soluble granule containing nitenpyram 10% was obtained.
[0013]
[Test Example(s)] The water-soluble granule manufactured by the examples 1-7 and the examples 1-2 of a comparison which were shown above was left for three months in the 40-degree C thermostat, the content of nitenpyram was measured with high performance chromatography, and the survival rate was computed by the degree type.
The survival rate (%) =(content / initial content) x100 result of nitenpyram was shown in [a table 1].
[0014]
[A table 1]
ニテンピラムの安定性

試　料	残存率（%）
実施例1	99.0
2	99.8
3	97.8
4	98.1
5	97.1
6	98.4
7	98.2
参考例1	91.1
2	90.3

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[A table 1] shows that the survival rate of NITEMUPIRAMU in the water-soluble granule obtained by this invention is clearly high compared with the water-soluble conventional granule. This shows that nitenpyram is stabilized in the water-soluble granule of this invention.
[0015]
[Effect of the Invention] The water-soluble granule of this invention can raise the conservation stability of a water-soluble agricultural-chemicals active ingredient, without spoiling soluble [to water].

[Translation done.]